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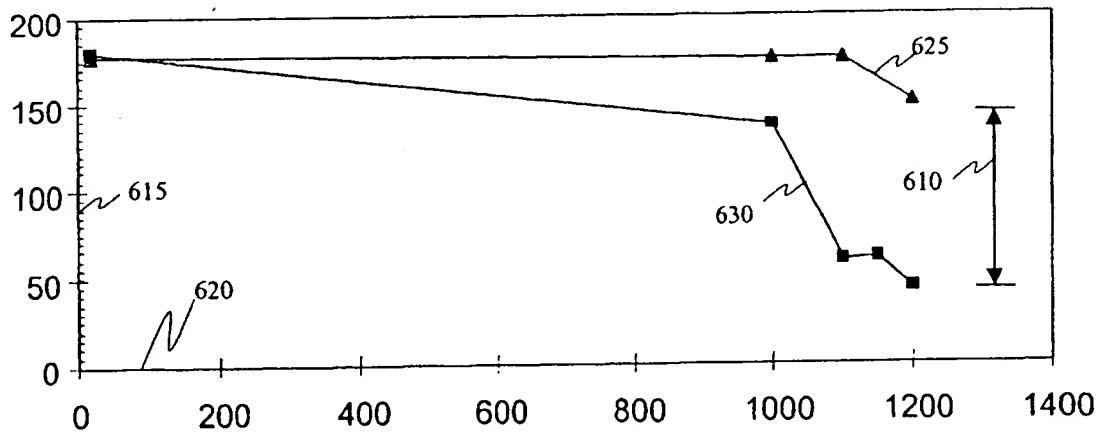
with international search report

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For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

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(54) Title: DAMAGE TOLERANT CMC USING SOL-GEL MATRIX SLURRY



WO 02/085618 A1

(57) Abstract: Disclosed are an oxide matrix composite that is stable for long-term exposures to temperatures of approximately 1,200° C and the methods of making the ceramic matrix composite, including wet lay-up (100), prepreg, and filament winding (510) fabrication methods. The oxide matrix composite can be made using commercially available refractory fibers that retain better than 85% of its original composite strength after 1,000 hours of exposure to such high temperature environments. The preferred alumina-based system demonstrates damage tolerance as relatively high strength retention properties and structural performance (610). The preferred refractory fibers are commercially available under the tradename of NEXTEL™ 720.

Nonprovisional Patent Application of

Steven Carl Butner and Thomas Barrett Jackson

for

TITLE: DAMAGE TOLERANT CMC USING SOL-GEL MATRIX SLURRY

CROSS-REFERENCE TO RELATED APPLICATION

This application claims priority from the following U.S. provisional Patent Application, the disclosure of which, including all appendices and all attached documents, is incorporated by reference in its entirety for all purposes: U.S. Provisional Patent Application Ser. No. 60/286,392, Steven Carl Butner and Thomas Barrett Jackson entitled, "DAMAGE TOLERANT CERAMIC MATIX COMPOSITE BY WET LAY-UP/PREPREG FABRICATION USING A SOL-GEL MATRIX," filed April 24, 2001.

175°C, and sinterable at nominal ranges of atmospheric pressure

9. A ceramic matrix composite as claimed in claim 8 wherein said reinforcing fiber is selected from a group consisting of NEXTEL 720 1500 Denier 8HS and NEXTEL 720 3000 Denier 8HS.

10. A ceramic matrix composite as claimed in claim 8, wherein said one or more fillers is fine alumina with an average particle diameter of 0.5 micron or less.

11. A ceramic matrix composite as claimed in claim 8, wherein said one or more fillers are fine alumina with an average particle diameter of 0.5 micron or less and coarse alumina with an average particle diameter greater than 0.5 micro and less than 1 micron.

12. A ceramic matrix composite as claimed in claim 8, wherein said reinforcement fibers are selected from a group consisting of NEXTEL 312, NEXTEL 550, NEXTEL 610, NEXTEL 720, and NEXTEL 720.

13. A ceramic matrix composite as claimed in claim 8, wherein said one or more fillers is a coarse mullite an average particle diameter greater than 0.5 micron and less than 1 micron.

14. A ceramic matrix composite as claimed in claim 8, wherein said one or more fillers are fine alumina with an average particle diameter of 0.5 micron or less and a

coarse mullite with an average particle diameter greater than 0.5 micron and less than 1 micron.

15. A method of forming an oxide-oxide ceramic matrix composite that comprises the steps of:
combining alumina sol and fine alumina thereby making a slurry;
prepping the slurry into an oxide fabric thereby making one or more prepreg plies;
staging each prepreg ply to about 80 to 98% of its original weight;
stacking the prepreg plies, one atop one another;
laminating the stacked plies using pressures of less than 100 psi and temperatures less than 175°C thereby making a laminated component; and
sintering the laminated component at a nominal range of atmospheric pressure thereby making an oxide-oxide ceramic matrix composite.

16. The method of forming an oxide-oxide ceramic matrix composite as claimed in claim 15 further comprising:
preceding the step of combining, the step of selecting alumina sol from the group consisting of aluminum hydroxylchloride, aluminum chloride hexahydrate, alpha aluminum monohydrate, aluminium oxide hydroxide aluminum hydroxide, and aluminum acetate.

17. The method of forming an oxide-oxide ceramic matrix composite as claimed in claim 15 wherein the alumina sol is colloidal and selecting is on the basis of surface areas ranging from 100 m²/g to 250 m²/g and average particle sizes ranging from 10 to 500 nanometers.

18. The method of forming an oxide-oxide ceramic matrix composite as claimed in claim 15 wherein the alumina sol is a solution yielding 8-30% weight percent alumina solids when heated to 1,200°C.

19. The method of forming an oxide-oxide ceramic matrix composite as claimed in claim 15 wherein the step of laminating is autoclaving.

20. The method of forming an oxide-oxide ceramic matrix composite as claimed in claim 15 wherein the step of laminating uses a lamination press.

21. The method of forming an oxide-oxide ceramic matrix composite as claimed in claim 15 wherein the step of laminating uses a compression mold whereby the plies are placed and laminated within the compression mold.

22. The method of forming an oxide-oxide ceramic matrix composite as claimed in claim 15, the method further comprising: (a) preceding the step of laminating, the step of affixing the plies to lamination tooling; and (b) preceding the step of sintering, the step of removing the laminated component from the lamination tooling.

23. The method of forming an oxide-oxide ceramic matrix composite as claimed in claim 22, wherein the step of combining alumina sol and fine alumina further comprises the step of combining coarse alumina with said alumina sol and said fine alumina.

24. The method of forming an oxide-oxide ceramic matrix composite as claimed in claim 22, wherein the step of combining alumina sol and fine alumina further comprises the step of combining coarse mullite with said alumina sol and said fine alumina.

25. The method of forming an oxide-oxide ceramic matrix composite as claimed in claim 22, wherein the step of combining alumina sol and fine alumina further comprises the step of combining diluted nitric acid with said alumina sol and said fine alumina.

26. The method of forming an oxide-oxide ceramic matrix composite as claimed in claim 22, wherein the step of combining alumina sol and fine alumina further comprises the step of combining with said alumina sol, and said fine alumina, organic processing aids selected from a group consisting of polyvinyl alcohol, methyl cellulose, propylene glycol, ethylene glycol and acacia gum.

27. The method of forming an oxide-oxide ceramic matrix composite as claimed in claim 22, wherein the oxide fabric is comprised of reinforcement fiber selected from a group consisting of NEXTEL 312, NEXTEL 550, NEXTEL 610, NEXTEL 650, and NEXTEL 720.

28. The method of forming an oxide-oxide ceramic matrix composite as claimed in claim 22, wherein the oxide fabric is comprised of NEXTEL 720 reinforcement fiber

29. The method of forming an oxide-oxide ceramic matrix composite as claimed in claim 22, wherein the step of

laminating is effected at pressures of less than 100 psi and temperatures less than 175°C.

30. A method of forming an oxide-oxide ceramic matrix composite that comprises the steps of:
combining alumina sol and fine alumina thereby making a slurry;
infiltrating an oxide fabric with the slurry thereby making one or more wet lay-up plies;
staging each wet lay-up ply to about 80 to 98% of its original weight;
stacking the wet lay-up plies, one atop one another;
laminating the stacked plies using pressures of less than 100 psi and temperatures less than 175°C thereby making a laminated component; and
sintering the laminated component at a nominal range of atmospheric pressure thereby making an oxide-oxide ceramic matrix composite.

31. The method of forming an oxide-oxide ceramic matrix composite as claimed in claim 30 further comprising:
preceding the step of combining, the step of selecting alumina sol from the group consisting of aluminum hydroxylchloride, aluminum chloride hexahydrate, alpha aluminum monohydrate, aluminium oxide hydroxide aluminum hydroxide, and aluminum acetate.

32. The method of forming an oxide-oxide ceramic matrix composite as claimed in claim 30 wherein the alumina sol is colloidal and selecting is on the basis of surface areas ranging from 100 m²/g to 250 m²/g and average particle sizes ranging from 10 to 500 nanometers.

33. The method of forming an oxide-oxide ceramic matrix composite as claimed in claim 30 wherein the alumina sol is a solution yielding 8-30% weight percent alumina solids when heated to 1,200°C.
34. The method of forming an oxide-oxide ceramic matrix composite as claimed in claim 30 wherein the step of laminating is autoclaving.
35. The method of forming an oxide-oxide ceramic matrix composite as claimed in claim 30 wherein the step of laminating uses a lamination press.
36. The method of forming an oxide-oxide ceramic matrix composite as claimed in claim 30 wherein the step of laminating uses a compression mold whereby the plies are placed and laminated within the compression mold.
37. The method of forming an oxide-oxide ceramic matrix composite as claimed in claim 30, the method further comprising: (a) preceding the step of laminating, the step of affixing the plies to lamination tooling; and (b) preceding the step of sintering, the step of removing the laminated component from the lamination tooling.
38. The method of forming an oxide-oxide ceramic matrix composite as claimed in claim 30, wherein the step of combining alumina sol and fine alumina further comprises the step of combining coarse alumina with said alumina sol and said fine alumina.

39. The method of forming an oxide-oxide ceramic matrix composite as claimed in claim 30, wherein the step of combining alumina sol and fine alumina further comprises the step of combining coarse mullite with said alumina sol and said fine alumina.

40. The method of forming an oxide-oxide ceramic matrix composite as claimed in claim 30, wherein the step of combining alumina sol and fine alumina further comprises the step of combining diluted nitric acid with said alumina sol and said fine alumina.

41. The method of forming an oxide-oxide ceramic matrix composite as claimed in claim 30, wherein the step of combining alumina sol and fine alumina further comprises the step of combining with said alumina sol, and said fine alumina, organic processing aids selected from a group consisting of polyvinyl alcohol, methyl cellulose, propylene glycol, ethylene glycol and acacia gum.

42. The method of forming an oxide-oxide ceramic matrix composite as claimed in claim 30, wherein the oxide fabric is comprised of reinforcement fiber selected from a group consisting of NEXTEL 312, NEXTEL 550, NEXTEL 610, NEXTEL 650, and NEXTEL 720.

43. The method of forming an oxide-oxide ceramic matrix composite as claimed in claim 30, wherein the oxide fabric is comprised of NEXTEL 720 reinforcement fiber

44. The method of forming an oxide-oxide ceramic matrix composite as claimed in claim 30, wherein the step of

laminating is effected at pressures of less than 100 psi and temperatures less than 175°C.

45. A method of forming an oxide-oxide ceramic matrix composite that comprises the steps of:
combining alumina sol and fine alumina thereby making a slurry;
wet winding oxide filament with the slurry about a fixture, thereby making one or more wet filament winding plies;
stacking the wet filament winding plies, one atop one another;
laminating the stacked plies using pressures of less than 100 psi and temperatures less than 175°C thereby making a laminated component; and
sintering the laminated component at a nominal range of atmospheric pressure thereby making an oxide-oxide ceramic matrix composite.

46. The method of forming an oxide-oxide ceramic matrix composite as claimed in claim 45 further comprising: preceding the step of combining, the step of selecting alumina sol from the group consisting of aluminum hydroxylchloride, aluminum chloride hexahydrate, alpha aluminum monohydrate, aluminium oxide hydroxide aluminum hydroxide, and aluminum acetate.

47. The method of forming an oxide-oxide ceramic matrix composite as claimed in claim 45 wherein the alumina sol is colloidal and selecting is on the basis of surface areas ranging from 100 m²/g to 250 m²/g and average particle sizes ranging from 10 to 500 nanometers.

48. The method of forming an oxide-oxide ceramic matrix composite as claimed in claim 45 wherein the alumina sol is a solution yielding 8-30% weight percent alumina solids when heated to 1,200°C.

49. The method of forming an oxide-oxide ceramic matrix composite as claimed in claim 45 wherein the step of laminating is autoclaving.

50. The method of forming an oxide-oxide ceramic matrix composite as claimed in claim 45 wherein the step of laminating uses a lamination press.

51. The method of forming an oxide-oxide ceramic matrix composite as claimed in claim 45 wherein the step of laminating uses a compression mold whereby the plies are placed and laminated within the compression mold.

52. The method of forming an oxide-oxide ceramic matrix composite as claimed in claim 45, the method further comprising: (a) preceding the step of laminating, the step of affixing the plies to lamination tooling; and (b) preceding the step of sintering, the step of removing the laminated component from the lamination tooling.

53. The method of forming an oxide-oxide ceramic matrix composite as claimed in claim 45, wherein the step of combining alumina sol and fine alumina further comprises the step of combining coarse alumina with said alumina sol and said fine alumina.

54. The method of forming an oxide-oxide ceramic matrix composite as claimed in claim 45, wherein the step of combining alumina sol and fine alumina further comprises the step of combining coarse mullite with said alumina sol and said fine alumina.

55. The method of forming an oxide-oxide ceramic matrix composite as claimed in claim 45, wherein the step of combining alumina sol and fine alumina further comprises the step of combining diluted nitric acid with said alumina sol and said fine alumina.

56. The method of forming an oxide-oxide ceramic matrix composite as claimed in claim 45, wherein the step of combining alumina sol and fine alumina further comprises the step of combining with said alumina sol, and said fine alumina, organic processing aids selected from a group consisting of polyvinyl alcohol, methyl cellulose, propylene glycol, ethylene glycol and acacia gum.

57. The method of forming an oxide-oxide ceramic matrix composite as claimed in claim 45, wherein the oxide fabric is comprised of reinforcement fiber selected from a group consisting of NEXTEL 312, NEXTEL 550, NEXTEL 610, NEXTEL 650, and NEXTEL 720.

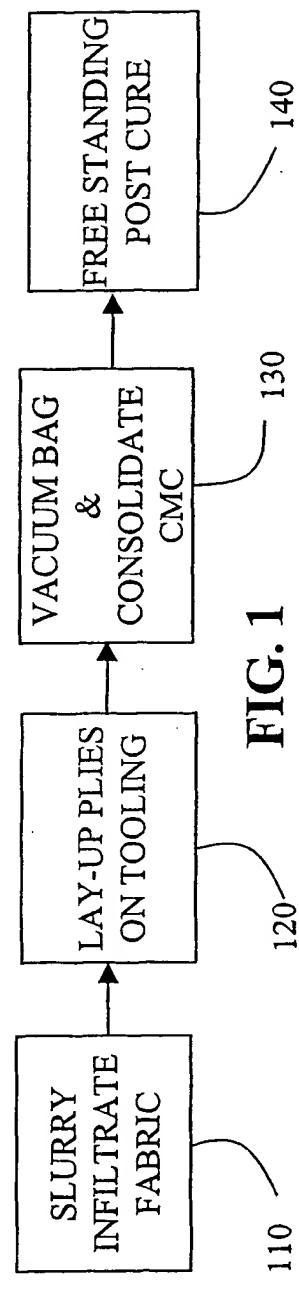
58. The method of forming an oxide-oxide ceramic matrix composite as claimed in claim 45, wherein the oxide fabric is comprised of NEXTEL 720 reinforcement fiber

59. The method of forming an oxide-oxide ceramic matrix composite as claimed in claim 45, wherein the step of

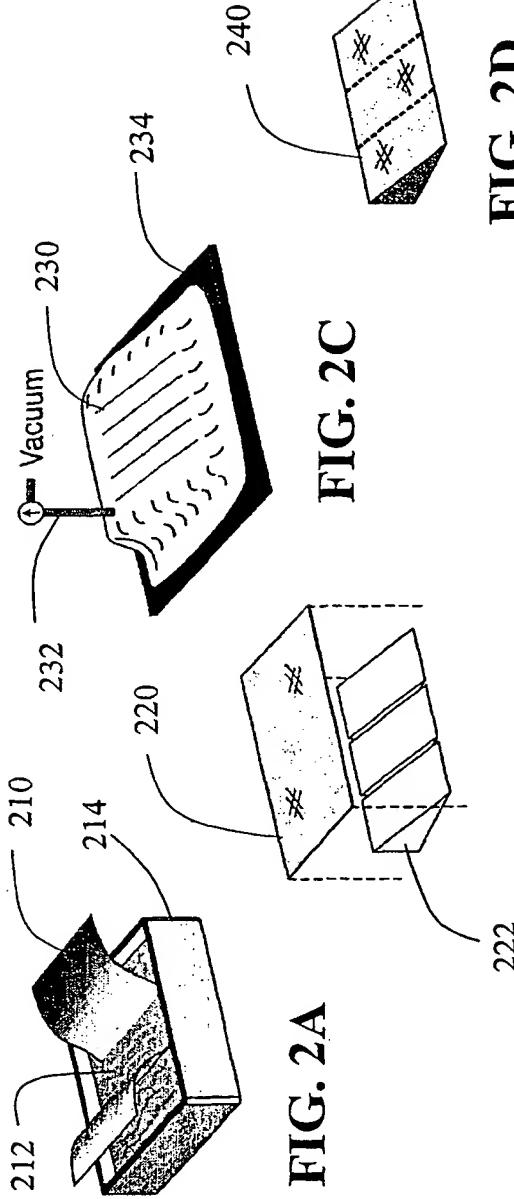
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laminating is effected at pressures of less than 100 psi and temperatures less than 175°C.

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100



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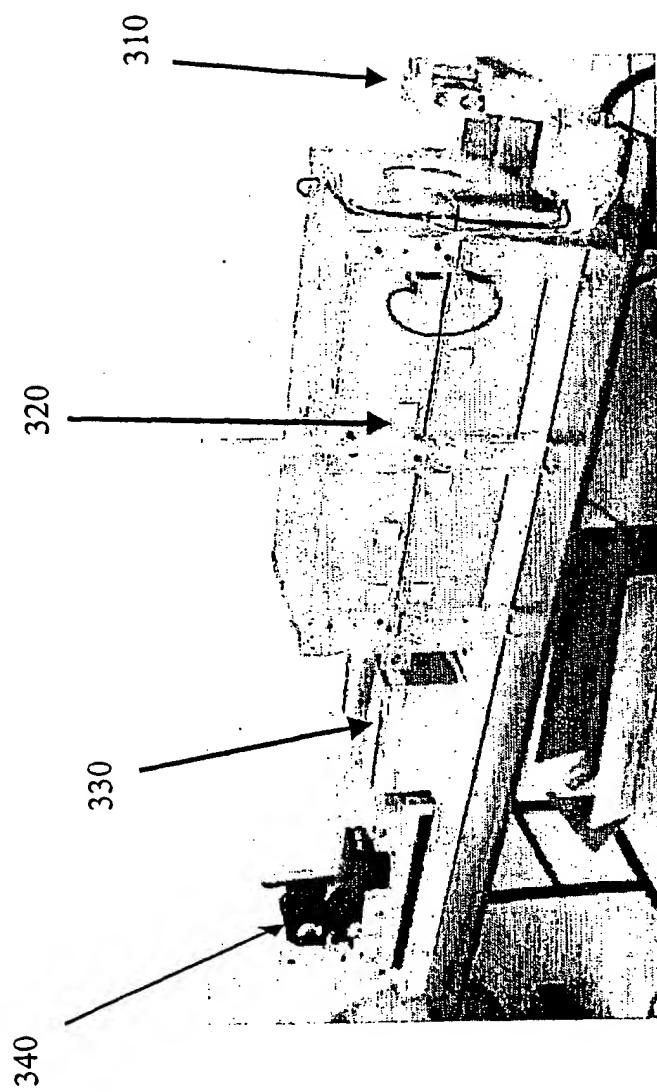
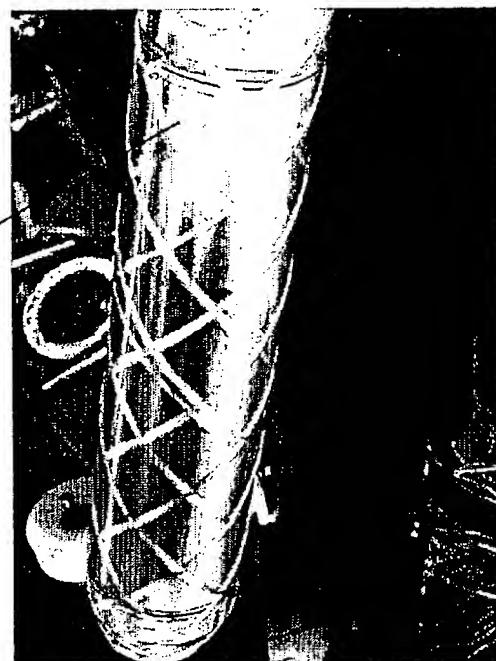


FIG. 3

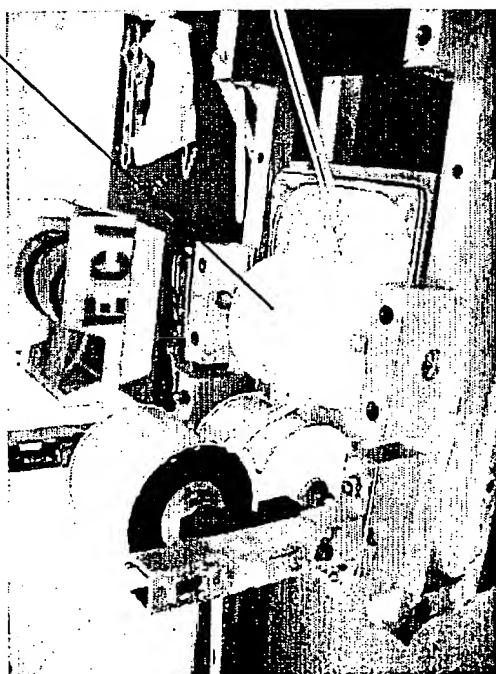
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FIG. 5



510

FIG. 4



340

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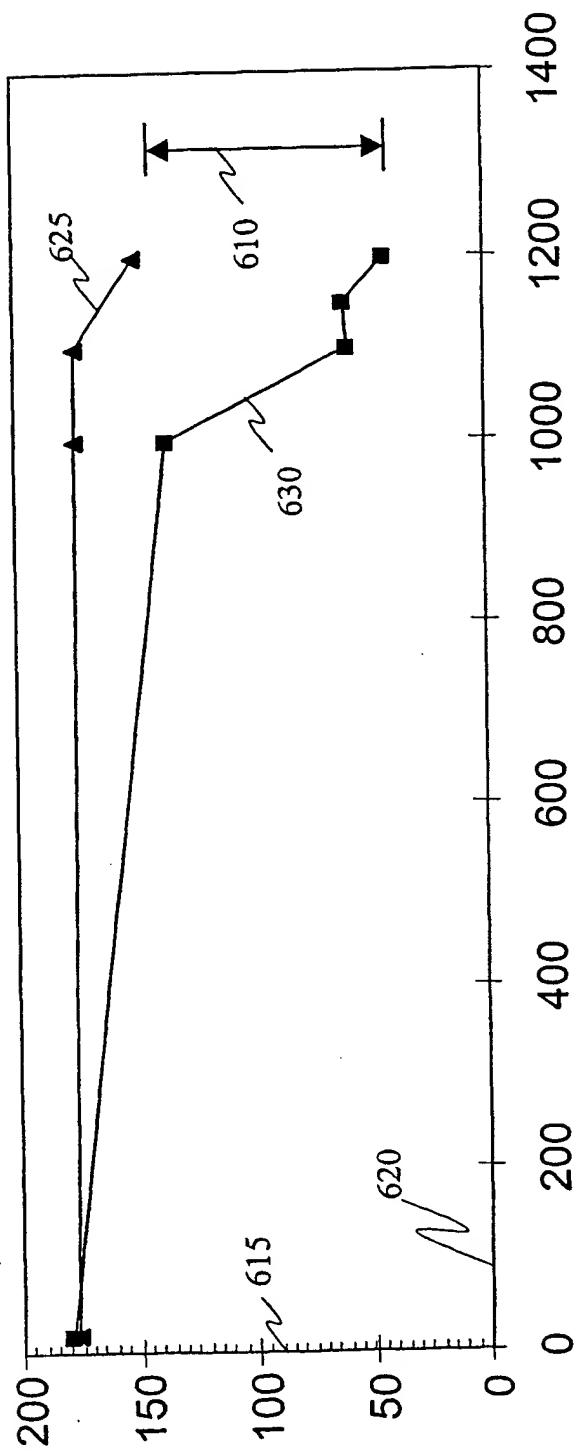


FIG. 6

INTERNATIONAL SEARCH REPORT

International application No.

PCT/US02/12601

A. CLASSIFICATION OF SUBJECT MATTER

IPC(7) :Please See Extra Sheet.

US CL :Please See Extra Sheet.

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 428/293.4;

427/389.8,386.

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched
~~TALEN~~

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EAST

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A,P	US 2002/0039644 A1 (KIMBARA et al.) 04 April 2002 see example 4.	1-14
A	US 6,117,516 A (NAKATA) 12 September 2000, see entire document.	15-59

 Further documents are listed in the continuation of Box C. See patent family annex.

"A"	Special categories of cited documents: document defining the general state of the art which is not considered to be of particular relevance	"T"	later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"E"	earlier document published on or after the international filing date	"X"	document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"L"	document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	"Y"	document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"O"	document referring to an oral disclosure, use, exhibition or other means	"&"	document member of the same patent family
"P"	document published prior to the international filing date but later than the priority date claimed		

Date of the actual completion of the international search

14 JUNE 2002

Date of mailing of the international search report

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INTERNATIONAL SEARCH REPORT

International application No.

PCT/US02/12601

A. CLASSIFICATION OF SUBJECT MATTER:

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A. CLASSIFICATION OF SUBJECT MATTER:

US CL :

428/293.4;

427/389.8,386.